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Orientations of recrystallization nuclei developed in columnar-grained Ni at triple junctions

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Abstract. A high purity columnar grained nickel sample with a strong <001> fiber texture was cold rolled to 50% reduction in thickness, followed by annealing at different temperatures. Optical microscopy was used to depict the grain boundaries prior to annealing and to detect nuclei formed on grain boundaries after annealing. Electron backscatter diffraction was performed to characterize the orientations of the nuclei and the deformed grains. Hardness tests were conducted at deformed grains. The potentials of triple junctions as preferential nucleation sites, the influence of deformation differences between adjacent grains on nucleation and orientation relationships between nuclei and parent matrix are analyzed.

1. Introduction

The nucleation process of recrystallization strongly affects the evolution of microstructure and texture and is thus of central importance for the properties of recrystallized materials [1, 2]. However, nucleation is difficult to study because of the small size of nuclei and the uncertainty about where the nuclei will form so that the investigation of nucleation is like a "needle in hay-stack" problem. Therefore, it is advantageous to focus on samples for which preferential nucleation sites may be active, such as materials with large particles [3, 4], with hardness indentations [5], scratches [6] and near triple junctions [7-9]. In the present study, we focus on nucleation at triple junctions and in particular on orientation relationships between the nuclei and their surrounding deformed matrix, where the nuclei are formed. The sample chosen for the investigation is high purity columnar grained nickel cold rolled to 50% reduction in thickness, thereby, nucleation from "below-the-surface deformed grains" is avoided.

2. Experimental

A high-purity columnar grained nickel sample with a strong <001> fiber texture was prepared by directional solidification. Viewed from the direction perpendicular to the grain growth direction, the material has a coarse grain structure with average grain diameter of 600µm, which is the same as the sample used in a previous study [9], and the grains have a perfect columnar shape. A sample was cold rolled to 50% reduction in thickness. During rolling, the grain growth direction is parallel to the normal direction (ND) of the sample. Three samples (named NO.1, NO.2 and NO.3) were cut from the rolled sheet. Each sample was cut perpendicular to the thickness direction into two identical pieces, the upper pieces for annealing experiments and the lower pieces for hardness tests. The samples were ground



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and electropolished. Extreme care was taken to get rid of surface imperfections, especially scratches, to avoid nucleation from such sites.

The upper pieces of the samples were first annealed at 350°C for one hour to somewhat recover the deformed microstructure, and then at 420°C, 400°C, and 395°C for one hour for sample No. 1, No.2, and No.3, respectively. After annealing, optical microscopy was used to reveal nucleation sites over the whole sample section. Electron backscatter diffraction (EBSD) mapping was then performed at the triple junctions where nuclei were found (using a Zeiss Auriga Dualbeam Station), to determine the orientations of the nuclei and the microstructures around the nuclei. Finally, microhardness tests were conducted using a force of 10g around triple junctions on the identical deformed lower pieces.

3. Results and discussion

3.1. Nucleation sites

A montage of optical micrographs of the deformed upper piece is used to show the grain boundaries for each sample, as demonstrated in Fig. 1 for sample NO.1. After annealing, nuclei are too small to be observed in a montage, so the nucleation sites are marked by red circles and numbered in the montage of deformed state. In total, 47 nucleation sites are observed in three samples: 35 right at triple junctions (e.g. nuclei 1-6 in Fig. 1), 12 at grain boundaries (e.g. nucleation site 7 in Fig. 1) and none within grain interiors. This result clearly shows the prevalence of triple junctions as the nucleation sites, which is in general agreement with previous investigations [7, 10]. It should, furthermore, be noted that all the nuclei formed at grain boundaries are very close to the triple junctions, within 50µm, and may well be formed close to the triple junctions at other section (although the grain boundaries are almost perpendicular to the RD/TD plane, there are of course some small variations).

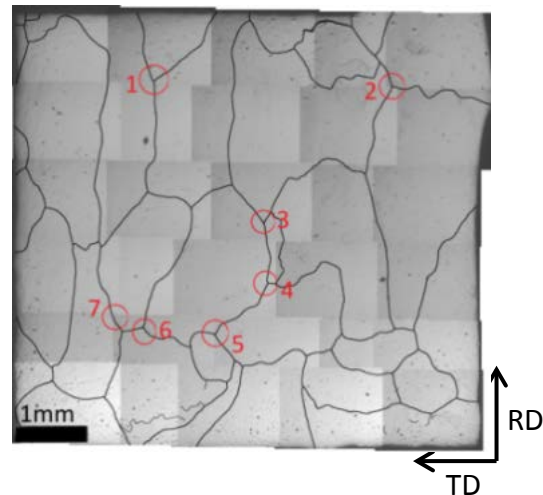


Fig.1 A montage of optical micrographs showing the grain boundary structure of the deformed upper piece of sample No.1. Numbers and red circles are used to identify nucleation sites of this sample after annealing.

3.2. Orientation relationship

The microstructure around 33 nucleation sites out of in total 47 were characterized using EBSD, including 22 at triple junctions and 11 at grain boundaries. Two examples are shown in Fig. 2, where nuclei and deformed grains are marked and numbered as Ni ($i = 1-2$.) and Gj ($j = 1-6$), respectively. Based on the misorientations between the nuclei and their surrounding deformed grains, the nuclei are divided into two groups. As seen in Fig. 2a, the nucleus N1 forms low angle boundaries to the deformed grain G1 at the triple junction, and its orientation lies at the outskirts of orientation cloud for grain G1 (see the pole figure). The nucleus N1 is therefore considered to originate from the deformed grain G1, and the nucleation is defined as parent type, Type I. A small grain, named T1, is also seen in Fig. 2a, which has a twin relationship with the nucleus N1. The grain T1 is therefore considered to develop by twinning during the growth of N1, and will not be considered as a new nucleus. In total Type I nucleation is found at 10 nucleation sites, including 8 at triple junctions and 2 at grain boundaries, where a deformed parent grain is found to be the same orientation with the nuclei. For these nuclei, subgrain growth or strain induced boundary migration could be the mechanisms for their formation.

It is found that there are 19 cases where the nuclei form high angle boundaries to the surrounding deformed matrix. But $\Sigma 3$ boundaries (following the Brandon criteria, i.e. 8.66° deviation from ideal $60^\circ \langle 111 \rangle$ relationship) can be found between the nuclei and one of their surrounding deformed grains. For example, in Fig. 2b the misorientation between the nucleus N2 and the deformed grain G6 is around $56^\circ \langle 443 \rangle$, which is about 8.1° away from

the ideal $60^\circ\langle 111 \rangle$. The nucleus N2 is therefore considered to originate from grain G6 through twinning. All these type of nuclei are defined as twin type, Type II. Twinning is a well-accepted mechanism for the nucleation of grains with new orientations. But it cannot be concluded from the present experiment whether the nucleus is formed by twinning directly from G6 or a nucleus Nx of parent type first forms below the surface and the nucleus N2 then forms by twinning during growth of the nucleus Nx. To answer this question, a full three dimensional characterization of the microstructure is necessary, which is planned for in the future.

At a few triple junctions, a series of nuclei are observed. These nuclei are often twin or double twin related to each other. For such cases the nuclei are classified as Type I and II if they are parent or twin related to any of the deformed grains respectively.

Finally, there are 4 cases where the nucleation does not belong to the two types. The nuclei do not form any $\Sigma 1$, $\Sigma 3$ or $\Sigma 9$ boundaries to any of the surrounding deformed grains. This result is in agreement with reference [9, 11], in which some of the recrystallized grains have new orientations and are not twin related to the deformation matrix. Compared with the results in Ref. [9], the present investigation shows a few nuclei with new orientations. This might be related to the recovery process occurring during the initial low temperature annealing in the present experiment. As the deformation structure coarsening and the stored energy is reduced during the recovery, one may speculate that the formation of nuclei with new orientation may be less likely because this formation will require energy to form the new high angle boundaries. At present, the mechanism of their formation is unclear.

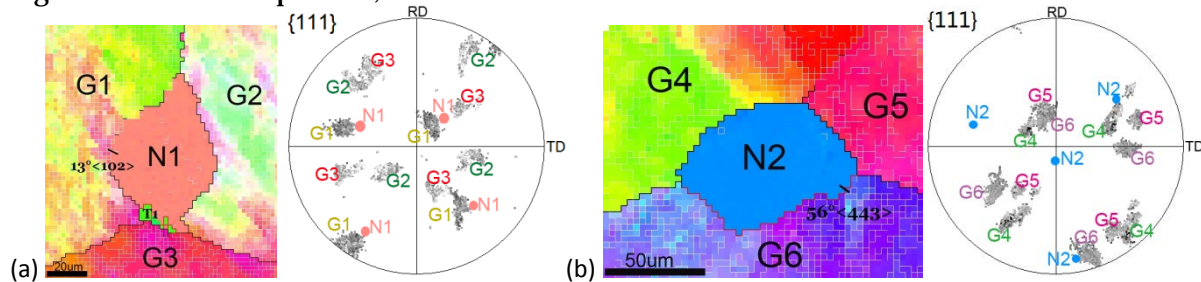


Fig. 2 EBSD orientation maps and $\{111\}$ pole figures to show the orientations of deformed grains and nuclei; Fine white lines show low angle boundaries ($\geq 1.5^\circ$ and $< 15^\circ$), coarse black lines high angle boundaries ($\geq 15^\circ$), and red lines and purple lines $60^\circ/[111]$ twin boundaries and $39.8^\circ[110]$ second-order-twin boundaries, respectively.

3. 3. Microhardness tests

An example of microhardness measurements at a triple junction is shown in Fig. 3a. In total microhardness around 21 triple junctions is measured, including 13 triple junctions where nuclei are found at the same triple junctions in the identical upper pieces, defined as Group A triple junctions, and 8 triple junctions where no nucleus is found in the identical annealed pieces, defined as Group B triple junctions. The microhardness distributions around triple junctions within both groups are shown in Fig. 3b; all three microhardness values of the grains at each triple junction are included. It is seen that the microhardness distribution of Group A triple junctions spans a wider range than that of Group B triple junctions. On average, however, the hardness values for Group A and B triple junctions are almost similar, namely 2.065 GPa and 2.072 GPa, respectively. Combining the orientation analysis in section 3.2, where nucleation sites are determined to be specific grains at the triple junctions, and the hardness for all three grains around these triple junctions, it is found that the nuclei can originate from crystals with either lowest or highest microhardness values at Group A triple junctions. The hardness value of the grain at a triple junction can therefore not be used to indicate if nucleation will occur at that triple junction or not.

That the distribution of microhardness of Group A triple junctions is clearly wider than that of Group B triple junctions, may however imply that larger differences in hardness between the grains at a triple junction may lead to nucleation. The difference between the maximum and minimum microhardness, ΔH_v , for each triple junction is therefore calculated and shown in Fig. 3c. It can be seen that the Group A triple junctions have a larger microhardness difference than the Group B triple junctions.

Nucleation by subgrain growth or strain induced boundary migration would be facilitated by a difference in stored energy (here taken proportional to the microhardness), which will drive the boundary towards the higher stored energy. It thus makes sense that more nucleation is seen at Group A than Group B triple junctions. It has, however, to be noticed that there are also Group A junctions with low ΔH_v values and that the statistics of the present investigation is poor. Also it is not clear if the initial recovery in the present work may influence the result. However it would be of interest to investigate a possible effect of ΔH_v on the nucleation potentials of triple junctions.

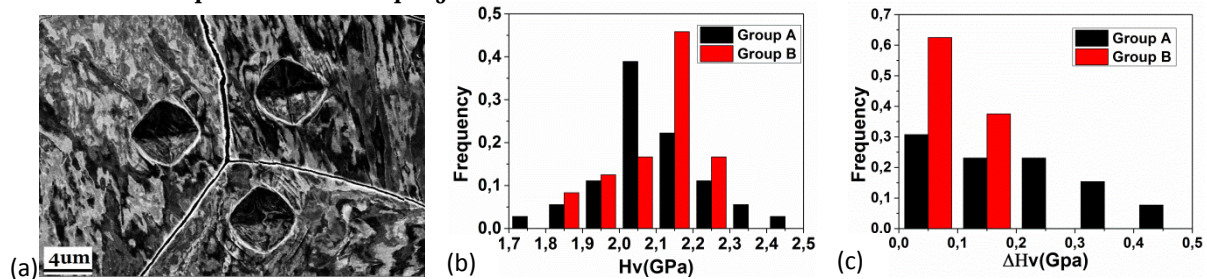


Fig. 3 (a) Example showing the hardness tests around a triple junction, and (b) the distribution of hardness and (c) ΔH_v values of the maximum and minimum hardness at Group A and Group B triple junctions.

4. Conclusions

In a cold rolled and recovered columnar grained pure Ni sample it is found that all nuclei form at triple junctions (74.5%) and at original grain boundaries (25.5%). The vast majority of the nuclei have parent orientations or are twin related to a parent deformed grains. Only at 4 nucleation sites, (12.1%) nuclei are formed with new orientations. This is lower than in a previous investigation and may be an effect of the initial recovery of the sample in the present investigation.

Microhardness measurements at triple junctions in the initial deformed sample indicate a weak correlation between the difference in hardness of the grains at the triple junctions, ΔH_v , and the potentials of the junction to form nuclei; the higher the difference, the more likely is nucleation. Further studies are needed to test this hypothesis.

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